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SPECIFICATION FOR 3,3′-DICHLOROBENZIDINE SALTS

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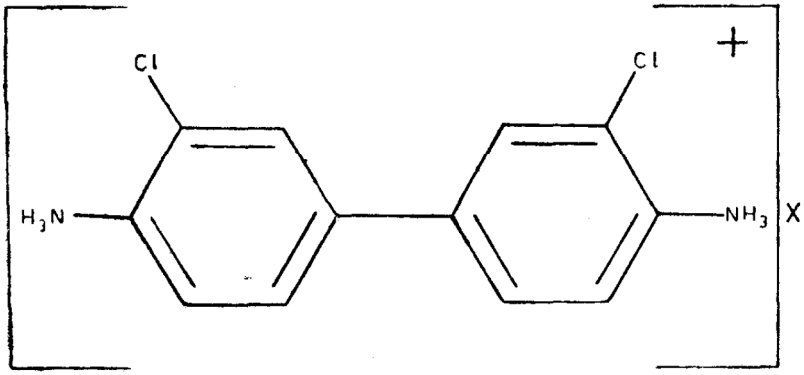
SPECIFICATION FOR 3,3'-DICHLOROBENZIDINE SALTS

0. FOREWORD

0.1 This Indian Standard was adopted by the Indian Standards Institutio n on 25 January 1985, after the draft finalized by the Dye Intermediate Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

0.2 3,3'-dichlorobenzidine is an important dye intermediate available as its dihydrochloride or monosulphate in the form of wet cake. It is used in the manufacture of organic pigments and dyes.

0.3 3,3'-Dichlorobenzidine and its salts are represented by the following structural formulae:



3,3'-DICHLOROBENZIDINE SALT

where

X = 2 Cl^- for dihydrochloride or SO_4^{--} for sulphate

i) 3,3'-Dichlorobenzidine dihydrochloride ($C_{12}H_{12}Cl_4N_2$)
(Molecular mass 326·1)

ii) 3,3'-Dichlorobenzidine sulphate ($C_{12}H_{12}Cl_2N_2O_4S$)
(Molecular mass 351·1)

0.4 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS : 2-1960*. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

1. SCOPE

1.1 This standard prescribes the requirements and the methods for sampling and test for 3,3'-dichlorobenzidine salts.

2. REQUIREMENTS

2.1 Description — The material shall be greyish-white moist powder free from extraneous impurities.

2.2 The material shall also comply with the requirements given in Table 1.

TABLE 1 REQUIREMENTS FOR 3,3'-DICHLOROBENZIDINE SALTS

Sl No.	Characteristic	Requirements for		Method of Test (Ref to Cl No. in Appendix A)
		Dihydrochloride	Sulphate	
(1)	(2)	(3)	(4)	(5)
i)	Assay (on dry basis) calculated as free base (molecular mass 253·1), percent by mass, *Min*	76·0	70·0	A-1
ii)	Matter insoluble on tetraazotization, percent by mass, *Max*	0·5	0·5	A-2
iii)	Acidity (in moles per mole of free base), *Min*	2·0 of hydrochloric acid	1·0 of sulphuric acid	A-3

3. PACKING AND MARKING

3.1 Packing — The material shall be packed in steel drums (*see* IS : 2552-1979†) lined with suitable polyethylene film or as agreed to between the purchaser and the supplier. Each container shall be securely closed.

*Rules for rounding off numerical values (*revised*).

†Specification for steel drums (galvanized and ungalvanized) (*second revision*).

3.2 Marking — Each container shall bear legibly and indelibly the following information:

a) Name of the material;
b) Name of the manufacturer and his recognized trade-mark, if any;
c) Free base, per cent by mass;
d) Moisture, percent by mass;
e) Batch number;
f) Tare, net and gross mass; and
g) The minimum cautionary notice worded as under:

'HAZARDOUS ! KEEP AWAY FROM DUST ! POISONOUS'
'AVOID INHALATION AND CONTACT WITH SKIN OR EYES'

3.2.1 The containers may also be marked with the ISI Certification Mark.

NOTE — The use of the ISI Certification Mark is governed by the provisions of the Indian Standards Institution (Certification Marks) Act and the Rules and Regulations made thereunder. The ISI Mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard under a well-defined system of inspection, testing and quality control which is devised and supervised by ISI and operated by the producer. ISI marked products are also continuously checked by ISI for conformity to that standard as a further safeguard. Details of conditions under which a licence for the use of the ISI Certification Mark may be granted to manufacturers or processors, may be obtained from the Indian Standards Institution.

4. SAMPLING

4.1 Representative samples of the material shall be drawn as prescribed in **3** of IS : 5299-1969*.

4.2 Number of Tests

4.2.1 Assay shall be done on each of the individual samples.

4.2.2 The remaining characteristics given in the specification shall be tested on the composite sample.

4.3 Criteria for Conformity — The lot shall be declared as conforming to the requirement of this specification if all the test results on each of the individual samples and the composite sample satisfy the relevant specification requirements, otherwise not.

*Methods of sampling and tests for dye intermediates.

5. TEST METHODS

5.1 Tests shall be carried out according to the methods prescribed in Appendix A, as indicated in col 5 of Table 1.

5.2 Quality of Reagents — Unless specified otherwise, pure chemicals and distilled water (*see* IS : 1070-1977*) shall be employed in tests.

NOTE — ' Pure chemicals ' shall mean chemicals that do not contain impurities which affect the results of analysis.

APPENDIX A

(*Table* 1, and *Clause* 5.1)

METHODS OF TEST FOR 3,3'-DICHLOROBENZIDINE DIHYDROCHLORIDE/SULPHATE

A-1. ASSAY

A-1.0 General — Dichlorobenzidine dihydrochloride/sulphate is usually supplied as moist powder. Percent purity shall be determined on dry sample.

A-1.1 Determination

A-1.1.0 *Outline of the Method* — It is determined by direct titration involving diazotization of the amine under acidic conditions with standard sodium nitrite solution using starch iodide paper as an indicator (external).

A-1.2 Reagents

A-1.2.1 *Sodium Nitrite Solution* — 0·2 N.

A-1.2.2 *Concentrated Hydrochloric Acid* — (*see* IS : 265-1976†).

A-1.2.3 *Potassium Bromide*

A-1.2.4 *Starch Iodide Indicator Paper*

A-1.3 Procedure — Weigh accurately about 1·0 g of the dry sample in a weighing bottle and transfer it into one-litre beaker. Add 10 ml of concentrated hydrochloric acid. Add 60 to 80 ml of distilled water with a measuring cylinder. Heat it on a hot plate and agitate until sample is dissolved. *Do not boil it.* Cool to 0 to 5°C by keeping in an ice-bath. Add 0·5 g of potassium bromide. Titrate with sodium nitrite solution making the additions dropwise towards the end of the titration until an immediate blue spot appears on starch iodide paper.

*Specification for water for general laboratory use (*second revision*).
†Specification for hydrochloric acid (*second revision*).

A-1.4 Calculation

$$\text{Assay (calculated as free base) (molecular mass 253·1), percent by mass} = \frac{V \times N \times 0{\cdot}126\,5 \times 100}{M}$$

where

V = volume in ml of sodium nitrite solution,

N = normality of sodium nitrite solution, and

M = mass in g of the sample taken for the test.

A-2. MATTER INSOLUBLE ON TETRA-AZOTIZATION

A-2.0 Outline of the Method — The sample is dissolved in an appropriate quantity of hydrochloric acid and tetra-azotized with sodium nitrite solution until tetra-azotization is complete. The resulting solution shall be clear and devoid of any insoluble matter.

A-2.1 Reagents

A-2.1.1 *Sodium Nitrite Solution* — 30 percent (*m/v*).

A-2.1.2 *Concentrated Hydrochloric Acid* — 30 percent.

A-2.1.3 *Starch Iodide Indicator Paper*

A-2.2 Procedure — Weigh accurately about 10 g of dry sample (on the basis of 100 percent freebase), transfer it to one-litre beaker and add 300 ml water. Keep the contents in rapid mechanical agitation. Add 30 ml of concentrated hydrochloric acid. Agitate for 15 minutes and then cool internally with sufficient amount of crushed ice to obtain a temperature of 0 to 5°C. Also cool it externally by keeping in water-ice mixture. Add at once from a burette 18·6 ml of 30 percent sodium nitrite solution through a long funnel with its tip dipped well below surface of the solution. Rinse funnel with ice cold water. Remove the funnel and continue agitation for 20 minutes at 0 to 5°C throughout. During this interval continually test the solution for excess nitrite with starch iodide indicator paper and excess acid with congo red indicator paper.

NOTE — If it so happens that a nitrite deficiency develops, immediately add 2 ml portion of 30 percent sodium nitrite solution and continue checking for excess nitrite, otherwise tetrazo of dichlorobenzidine immediately forms a dark insoluble matter through coupling reaction. If there is any appreciable amount of insoluble matter, filter solution through a tared gooch crucible (M_1). Wash beaker with several portions of hydrochloric acid (1 : 10) and add washing to gooch. Finally wash residue in gooch crucible with 50 ml of hydrochloric acid (1 : 10). Suck dry and place in the oven at 100°C for 2 hours and then cool in a desiccator. Weigh and repeat the process until constant mass is obtained (M_2).

A-2.3 Calculation

$$\text{Matter insoluble on tetra-azotization, percent by mass} = \frac{(M_2 - M_1)}{M_3} \times 100$$

where

M_1 = initial mass in g of the crucible,
M_2 = final mass in g of the crucible, and
M_3 = mass in g of the sample taken for the test.

A-3. DETERMINATION OF ACIDITY

A-3.1 Outline of the Method — The test sample is hydrolyzed with excess alkali and this excess alkali is back titrated against standard acid.

A-3.2 Reagents

A-3.2.1 *Sodium Hydroxide Solution* — 1 N

A-3.2.2 *Hydrochloric Acid Solution* — 1 N.

A-3.2.3 *Phenolphthalein Indicator Solution*

A-3.3 Procedure — Weigh accurately about 1 to 1·5 g of sample on the basis of 100 percent free base and transfer it into 250 ml conical flask. Add 20 ml of sodium hydroxide (1 N) by means of pipette and about 50 ml of distilled water. Heat the content of the flask till sample undergoes complete hydrolysis. Cool the flask at room temperature and titrate it against hydrochloric acid (1 N) using phenolphthalein as an indicator.

A-3.4 Calculation

Acidity (in moles per mole of free base)

A-3.4.1 *For Hydrochloride*

$$= \frac{(V_1 N_1 - V_2 N_2) \times 0{\cdot}253\ 1}{M}$$

A-3.4.2 *For Sulphate*

$$= \frac{(V_1 N_1 - V_2 N_2) \times 0{\cdot}253\ 1}{M \times 2}$$

where

V_1 = volume in ml of sodium hydroxide added,
N_1 = normality of sodium hydroxide,
V_2 = volume in ml of hydrochloric acid for titration,
N_2 = normality of hydrochloric acid, and
M = mass in g of the sample expressed as 100 percent free base.